### REMARKS

Claims 14-17, 19-36 are pending in this application. Claims 37-38 are cancelled, without prejudice or disclaimer, to simplify this application. Claims 27, 29, and 33 are characterized in the final office action (page 7) as being allowable if rewritten in independent form. Claims 14, 17, 36 have been amended above. The amendment to claim 14 merely makes express what was already identically understood by "it" and is only for clarity and simplicity of communication, with no change in meaning. As to the amendment to claim 36, see, e.g., Applicant's specification at page 1, line 7 and page 17, lines 18-19.

At the beginning of the office action, the Examiner asks about the foreign priority document. There is a European parallel application, and the EPO has the priority document on file (open to file inspection for the public). Please see <a href="https://www.epoline.org/portal/public">www.epoline.org/portal/public</a>, click: "Register plus", search:

"Veroffentlichungsnummer" EP1460095, click lefthand: "Alle Dokumente", mark the box for "04/03/2004 Priority Document" and open the document. The priority document is in German.

At paragraph 2 of the office action, Claim 17 has been objected to as being of improper dependent form for failing to further limit the subject matter of a previous claim. To advance prosecution, Claim 17 has been amended above.

At paragraph 3 of the office action, Claim 31 has been rejected under 35 U.S.C. 112, first paragraph, as failing to comply with the written description requirement. At paragraph 4 of the office action, Claim 31 also has been rejected under 35 U.S.C. 112, second paragraph, as being indefinite.

Claim 31 satisfies the written description requirement. See page 15, original claim 3, of Applicants' specification.

Claim 31 is not indefinite. The claim refers to component (B) and claim 21, where the compound containing hydrogen active in a polyurethane reaction is selected from polyols. Mixtures of SH and OH or NH and OH are not possible here. The percentage (60%) is an absolute percentage, i.e. 60% of all OH-groups are primary OH-groups.

Reconsideration and withdrawal of the rejections based on 35 U.S.C. 112 relating to Claim 31 are respectfully requested.

At paragraph 7 of the office action, Claims 17 and 36-38 have been rejected under 35 U.S.C. 102(b and e) as being anticipated by Schumann. The Examiner states that "Claims 17 and 36 recite 'comprising' regarding the foil and therefore encompass the adhesive layer of the reference."

Applicants respectfully traverse the anticipation rejection as follows.

As a preliminary matter, the US '243 published patent application mentioned by the Examiner cannot be cited as a reference herein because that application was filed April 25, 2005 which is <u>not</u> before Applicants' present U.S. application filing date of March 4, 2004 (and their foreign priority date of March 18, 2003). Please note that the '243 application was a continuation<u>-in-part</u> and clearly has much new matter, as may be seen just by looking at the figures where figures 2-7B were added in the '243 U.S. patent application that were not present in the German-language document EP '993. The availability of the Englishlanguage 09/698,404 document makes the too-late '243 document wholly irrelevant. For the Examiner's convenience, 09/698,404 is attached hereto.

The Examiner's position is that because Claim 36 recites "comprising", an adhesive layer can be present. Claim 36 as amended is clearly a foil that is "a layer" which means that multi-layer structures (which is what Schumann discloses) are clearly excluded. Applicant's Claim 36 requiring that the foil be a single layer is distinguished from Schumann's multi-layer laminates.

Reconsideration and withdrawal of the anticipation rejection are respectfully requested.

At paragraph 8 of the office action, Claims 14-17, 19-26, 28, 30-32, and 34-38 have been rejected under 35 U.S.C. 103(a) as being unpatentable over each of EP '993 and US Pat. Application Pub. No. US 2005/0263243. The Examiner states that "Removal of the film, i.e foil, of the reference from its release layer constitutes the 'demolding' of the instant claims."

Applicants respectfully traverse this obviousness rejection.

Regarding Applicant's claim 14 to a process, Schumann EP '993 fails to

<sup>&</sup>lt;sup>1</sup>As discussed above, as a preliminary matter, the US '243 CIP publication should not be used against the present application because of its too-late date. The Examiner's assumption that the '243 CIP was a fair translation of EP '993 is clearly not borne out by the fact that the '243 CIP has many added figures which were not present in EP '993.

disclose steps of "spraying the composition in one or more passes onto a smooth surface or into a mold, permitting the sprayed composition to react to completion, and demolding the foil after curing" as recited in Applicant's claim 14. Schumann EP '993 fails to teach "spraying." Also, Schumann EP '993 fails to teach a sprayed composition being permitted to react to completion. Whatever reacts in Schumann EP '993 is not a sprayed composition as in Applicants' claim 14.

Applicants' spraying is substantially different from what is disclosed in Schumann EP '993. Moreover, a demolding step following a spraying step is not suggested by nor obvious to a person of ordinary skill in Applicants' art from Schumann EP '993.

Also, Schumann EP '993 fails to disclose spraying the composition "onto a smooth surface or into a mold" as in Applicants' claim 14. By contrast, Schumann EP '993 applies the polyurethane composition to a backing material which is coated with a pressure-sensitive adhesive composition, see, e.g., Schumann 09/698,404 page 4 paragraph c).

Also, Applicants' claim 14 requires "a freely demoldable foil" in which the foil is clearly made from the polyurethane composition. Schumann EP '993 contains no teaching that the part of his article which contains the polyurethane composition is freely removable from anything; rather, it is another part (an adhesive surface) of Schumann EP '993's article that separates. Schumann EP '993 lacks any teaching or disclosure about producing a polyurethane article from a mold or smooth surface "freely demoldable", i.e., with the foil being required not to adhere to the mold and to keep its shape without danger of ruptures stretches.

Applicants' independent claim 36 – reciting "a <u>demolded</u> polyurethane foil consisting of a sprayed foil or a cast foil with a layer thickness from 0.1 to 5 mm, which comprises a fine-particle oxide of a metal or of a metalloid, and which is free from amine initiators" – also is substantially distinguished from Schumann EP '993. It is not the polyurethane part of the Schumann article that detaches from something, but rather, the adhesive part. In Schumann, the polyurethane part stays attached and as it is, therefore the polyurethane part is closer to being the opposite of "demolded."

Applicants' claim 36 is not obvious over Schumann EP '993 for the further

reason that Schuman's structure is multi-layer, not a demolded polyurethane foil with a layer thickness as in Applicant's claim 36. A sprayed foil or a cast foil as recited in Applicant's Claim 36 cannot be Schuman's multi-layer laminate.

Applicant's claims 14 and 36 are farther from Schumann EP '993 than the Examiner has recognized. In the office action, flawed and artificial assumptions have been set forth to seem to bring Applicants' claimed invention closer to Schumann but which are not fair assumptions for the following reasons. For example, in the office action (page 5), the assumption underlying the obviousness rejection is set forth that: "Removal of the film, i.e., foil, of the reference from its release layer constitutes the 'demolding' of the instant claims." This assumption is not valid, because in Schumann after curing the release paper can be pulled off for use from the adhesive, not from the cured polyurethane, therefore in Schumann the pulling off of the release paper cannot fairly be treated as "demolding" a polyurethane foil because the release paper is not being pulled-off from Schuman's polyurethane-containing backing but rather from the other surface, the adhesive surface.

In Schuman, if the first backing material is not a dehesive medium, the layer of this first backing material is not removed at all. In that case, the order of layers in Schumann is: (pressure-sensitive adhesive, first backing, polyurethane) or (pressure-sensitive adhesive, first backing, polyurethane, pressure-sensitive adhesive) – see Schumann 09/698,404 page 5, line 4 etc.; see also page 3, line 29 page 4. From these parts of Schuman, it is clear to see that Applicants' claimed inventive process is quite removed from what Schumann was doing and that a person of ordinary skill in Applicants' art would lack motivation to modify Schumann in the direction of Applicants' invention. Applicants especially note that an important advantage of the method of Schumann "is to be able to achieve effective anchoring between the polyurethane backing material and the incoming, web-form materials, i.e., for example, the pressure-sensitive adhesive composition." Schumann, 09/698,404, sentence bridging pages 10-11. Thus, the "anchoring" between the PU and the adjacent layer(s) is an important feature of Schumann (already following from the term "backing", implying to form a basis for some other material on top). At the same time it follows that it cannot be obvious to a person of ordinary skill in Applicants' art to demold Schumann's

backing alone as a single layer film. The result of making the assumption set forth in the office action would be to destroy the functionality of Schumann's article and make it unuseable for its intended purpose, for no reason. The assumption on which the obviousness rejection was made having been flawed, the rejection cannot be maintained.

For simplicity and brevity, remarks are not made for each dependent claim that has been rejected.

Wherefore, reconsideration and withdrawal of the art rejections are respectfully sought.

In view of the foregoing, it is respectfully requested that the application be reconsidered, that claims 14-17, 19-36 be allowed, and that the application be passed to issue.

Should the Examiner find the application to be other than in condition for allowance, the Examiner is requested to contact the undersigned at the local telephone number listed below to discuss any other changes deemed necessary in a telephonic or personal interview.

A provisional petition is hereby made for any extension of time necessary for the continued pendency during the life of this application. Please charge any fees for such provisional petition and any deficiencies in fees and credit any overpayment of fees to Attorney's Deposit Account No. 50-2041 (Whitham, Curtis, Christofferson & Cook, P.C.).

Respectfully submitted,
MMY G Gould

Mary E. Goulet Reg. No. 35,884

Whitham, Curtis, Christofferson & Cook, P.C. 11491 Sunset Hills Road, Suite 340 Reston, VA 20190

Tel. (703) 787-9400 Fax. (703) 787-7557

Customer No.: 30743

# Bei rsdorf Akti ngesellschaft Hamburg

5

# Description

# Process for continuous manufacture of self-adhesive articles by coating incoming web-form materials with two-component polyurethanes

10

15

The present invention relates to a process for the continuous production of self-adhesive articles such as, for example, self-adhesive tapes by coating an incoming material in web form or two simultaneously incoming web-form materials, disposed with their surfaces parallel above but not in contact with one another, with a reactive, two-component polyurethane backing material, at least one of the incoming web-form materials having been treated with a pressure-sensitive adhesive composition (self-adhesive composition).

Single-sided self-adhesive articles comprise at least two layers (laminae), namely the backing layer, which is not self-adhesive, and the pressure-sensitive adhesive composition applied to it. A double-sided self-adhesive tape is generally composed of at least three layers, namely the backing layer and the pressure-sensitive adhesive layers applied to it on both sides. Exceptions are double-sided self-adhesive articles wherein backing and adhesive layer are identical (known as single-layer products).

25

30

35

The mechanical properties of an adhesive tape (for example, tensile strength, extensibility, elasticity) are essentially determined by the backing. The backing, moreover, largely determines the optical properties of an adhesive tape (transparency, color) and, in the case of a single-sided self-adhesive article, the surface properties of the side which is not self-adhesive (texture, roughness, surface tension). The backing material is also a co-determinant of the adhesion properties of a self-adhesive article.

Appropriate backing materials include all materials in web form, for example papers, wovens, nonwovens, films or elastomers, each with different thicknesses, textures and polymer compositions.

30

35

5

10

In combination with the respective backing that is used, the adhesive layer critically determines the adhesion properties of a self-adhesive article, which are manifested, inter alia, in shear stability times, bond strengths, tip-shear behavior, peel increase behavior, redetachability, et cetera.

The base polymers of modern pressure-sensitive adhesives include natural and synthetic rubbers, polyacrylates, block copolymers with polystyrene block fractions, polyethylene-vinyl acetates and polyurethanes, which are usually used in combination with additives such as resins and plasticizers and/or other auxiliaries such as, for example, antioxidants, UV stabilizers or rheological additives.

The widespread general process for producing adhesive tapes comprises coating a separately produced, web-form backing material with an adhesive composition. The coating operation is normally carried out from a solution, i.e., the pressure-sensitive adhesion composition is converted to a spreadable consistency, using solvents, prior to coating.

Alternatively, and depending on the polymer composition, coating may also be effected from the melt, without solvent, in an extrusion process. This process has become established in particular in the case of pressure-sensitive adhesives based on thermoplastic elastomers.

Moreover, the assembly of backing and pressure-sensitive adhesive composition may also be produced by first applying the pressure-sensitive adhesive composition to a dehesive medium and subsequently applying it to the backing in a laminating process.

The coating of web-form backing materials with pressure-sensitive adhesive compositions is very well established as a process for producing self-adhesive articles.

Nevertheless, there are a number of fundamental disadvantages which are disruptively manifested in particular in the case of adhesive tapes having high or very specific profiles of requirements. For instance, in many cases the anchoring of the adhesive composition on the backing is a problem and requires a further process step, namely coating with a primer (pre-coat). In the case of a double-sided adhesive tape, of course, this must be

done on both sides, so resulting in a five-layer product structure. Moreover, there are occasionally no tailor-made backings available on the market, as are required for specific applications. In that case, either recourse is had to composite systems comprising individual backings, a further disadvantage of which is that they have to be joined using, for example, primer and adhesive systems, or else additional auxiliary layers are applied (for example, barrier layers preventing the migration of ingredients from the backing into the adhesive layer, or mirror layers for smoothing rough backing surfaces). All this results in increased complexity in the production process, and, ultimately, increased production costs.

10

In the text below, by way of example, a number of adhesive tapes from the comprehensive prior art are depicted, specifically those where polyurethanes or polyurethane films are used as the backing material.

15

WO 86/00536 A1 discloses a laminate comprising a polyurethane and a pressuresensitive adhesive layer, the laminate being used for a pellet package and, respectively, administration form. A polyurethane film, without further treatment, is provided with a selfadhesive coating which envelopes a pellet and at the same time is bonded with the pellet to the skin of the user.

25

30

US 5,127,974 A mentions a laminate comprising a polyurethane film with a self-adhesive coating. This laminate is used especially for the temporary protection of coated automobile surfaces.

DE 196 14 620 A1 and DE 197 33 014 A1 disclose a pressure-sensitive adhesive tape, coated on both sides with adhesive compositions, whose backing is formed by a formulated, crosslinked, unfoamed polyurethane. Formulation constituents of the backing are a crosslinked, unfoamed polyurethane, fillers,

and, if desired, further auxiliaries.

According to DE 196 14 620 A1, the polyurethane content of the backing is up to 50% by weight, preferably from 30% by weight to 40% by weight, the polyurethane being plasticizer-free. The fillers account for from 50% by weight to 70% by weight of the backing.

15

20

25

According to DE 197 33 014 A1, the polyurethane content of the backing is up to 50% by weight, preferably from 10% by weight to 40% by weight. The fillers account for from 40% by weight to 70% by weight of the backing, while the plasticizers and resins together are used at from 5% by weight to 30% by weight, in particular from 10% by weight to 25% by weight.

It is an object of the present invention to provide a process with which self-adhesive articles may be produced continuously without the need to produce separately the webform backing material of the self-adhesive article and then coat it with an adhesive composition, directly or by a laminating process, so that the fundamental disadvantages of the conventional production processes for adhesive tapes are unable to occur in the form. The backing is to be distinguished by a profile of properties which can be adjusted variably and diversely.

This object is achieved by a process as specified in the main claim. The subclaims relate to advantageous embodiments of the process. Finally, the concept of the invention also embraces self-adhesive articles produced by the process of the invention.

The invention accordingly provides the process described below for continuous production of self-adhesive articles whose backing comprises a polyurethane as base polymer.

The process of the invention is composed of the following individual steps:

- a) Essentially one polyol component is placed in a container A and essentially one isocyanate component is placed in a container B.
- b) The polyol component and the isocyanate component are mixed in a mixer.
- c) The polyurethane composition thus mixed is applied to a backing material which is coated with a pressure-sensitive adhesive composition and moves preferably at constant speed.
- 30 d) The laminate, comprising backing material, pressure-sensitive adhesive composition and polyurethane composition, is passed through a heat tunnel, in which the polyurethane composition cures.
  - e) The laminate is finally wound in a winding station.

10

15

20

25

In a preparation step, the backing material is treated on both sides, or in particular on one side, with a pressure-sensitive adhesive composition. This treatment takes place in a customary coating process, either from a solution or from the melt.

The backing material may be a dehesive medium, for example, a release paper or a release film. Alternatively, it may comprise any desired other backing material, for example, a paper, a woven, a nonwoven, a film or an elastomer, which after the last process step constitutes part of the overall backing of the self-adhesive article.

Where the backing material is a dehesive medium, the polyurethane composition is applied to the backing material coated with a pressure-sensitive adhesive composition, said application taking place such that the polyurethane composition is present on the pressure-sensitive adhesive composition.

Where the backing material is not a dehesive medium, the polyurethane composition is applied preferably to that side of the backing material on which there is no pressure-sensitive adhesive composition. Alternatively, however, in this case as well the polyurethane composition may be applied to the pressure-sensitive adhesive composition. If so, a further layer of adhesive should be applied to the outer face of the backing material.

In another preferred embodiment of the process, a second backing material is supplied at preferably constant speed to the polyurethane composition of the laminate.

In another preferred embodiment of the process, the second backing material has been treated with a pressure-sensitive adhesive composition.

In this case, an additional preparation step is necessary, in which the first preparation step is repeated accordingly, neither the pressure-sensitive adhesive composition nor the web-form material necessarily being identical with those from the first preparation step. If desired, after the heating tunnel, the second backing material is peeled off.

Also advantageous are further containers upstream of the mixer, containing catalysts, plasticizers, dyes and other additives, which may be introduced and added.

Coating with the reactive, two-component polyurethane onto the first backing material takes place preferably on a standard coating unit for the production of adhesive tapes.

Figure 1 depicts such a unit in a preferred embodiment.

35

30

20

25

30

The unit 100 possesses two bale unwinders 11, 12 for the incoming materials 1 and 2, and also a product winding station 21 and a bale winder 22 for any auxiliary material 3 that may become uncovered. Moreover, the unit 100 has a heating tunnel 31 in which the polyurethane composition 4 is cured.

The incoming, web-form materials 1 and 2 are guided so that the coating of the polyurethane composition 4 may take place directly in the gap between the two materials 1 and 2. The gap width is variable and freely adjustable.

Behind the gap, a web guide, not shown here, for the second backing material 2, via a belt, is advantageous with regard to the achievement of a good constancy of thickness of the polyurethane backing material 4.

In the case of a single-sided self-adhesive article, the upper bale unwinder and the bale winder for the auxiliary material 3 to be uncovered may, if appropriate, be omitted.

Alternatively, a dehesive material may be used whose function is merely to keep the shaft and the reactive polymer from coming into contact with one another, in order to avoid instances of curing of the polyurethane on the shaft.

The reactive polyurethane backing composition 4 is prepared continuously, directly before its application, from two components which react chemically with one another, namely a generally preformulated polyol component (A) and an isocyanate component (B), both of which are present in containers 41 and 42, respectively, preparation taking place in a mixing head (dynamic mixer) or a mixing pipe (static mixer) of a standard two-component mixing and metering unit 43, and are applied directly between the incoming, web-form materials 1 and 2.

A suitable two-component mixing and metering unit 43 is any corresponding standard commercial unit which is suitable for casting and is designed for short pot lives of generally less than one minute. It may be actualized either in a static system or else in a dynamic mixing system. In order to be able to coat the full width of the incoming materials 1 and, if appropriate, 2 with the polyurethane backing material 4, it is advantageous to install the mixing head or the mixing pipe such that it is movable on a traversing device, which then permanently travels the width of the incoming materials, in an oscillating manner.

30

35

5

10

In the subsequent heating tunnel section 31, the reactive polyurethane composition 4 cures to the desired backing material and, in general, attaches chemically to the incoming materials, i.e., in particular, adhesive coatings. Attachment to an acrylate takes place, for example, through the formation of a carboxamide. After the tunnel section 31, the finished product is wound up.

The heating tunnel section 31 is held preferably at temperatures between 20°C and 120°C.

In the case of a double-sided adhesive tape, a three-layer product structure is obtained, comprising adhesive composition, polyurethane backing, and adhesive composition. There is no need for a primer, nor for any other additional layer. The backing thickness is easily adjusted by way of the gap width on the applicator unit. Since the polyurethane components (A) and (B) contain no solvent, even very thick backings can be produced without bubbles using this process.

In one possible embodiment, the backing has a thickness of from 0.1 to 50 mm, preferably from 0.4 to 20 mm. The adhesive composition preferably has an application weight of from 10 g/m $^2$  to 100 g/m $^2$ .

Suitable polyurethane backing materials are all materials which comprise a polyurethane as base polymer and may be prepared in a two-component mixing process. The diversity of polyurethane chemistry, resulting both from the fullness of the polyurethane building blocks provided by the chemical base-materials industry and from the diverse possibilities of compounding with fillers, resins, plasticizers, other polymers, for example, epoxides, acrylates, natural and synthetic rubbers, ethylene-vinyl acetates, block copolymers with polystyrene block fractions, and other additives such as aging inhibitors, UV stabilizers or rheological additives, for instance, makes it possible to use this process to provide self-adhesive articles tailored to many fields of application.

Also suitable as the polyurethane backing material are all foamed materials which comprise a polyurethane as base polymer and may be prepared in a multicomponent mixing process. The foam structure may be achieved either chemically, for example, by means of an isocyanate/water reaction initiated during the mixing operation, or by blowing agents, or else physically, by the introduction of a gas (for example, nitrogen or air). The gas may be introduced both during the preparation of the A or B component and directly at the mixing head of a multicomponent mixing and metering unit. The gas

10

introduced into the mixing head represents, so to speak, the special case of a third component.

Express reference may be made to the depiction of the state of the art polyurethane chemistry in "Kunststoff-Handbuch" 7, Polyurethane, Becker/Braun (1993).

One possible embodiment of the backing comprises as formulation constituents a crosslinked, unfoamed polyurethane, fillers, and, if desired, further auxiliaries.

The polyurethane content of the backing is up to 50% by weight, preferably from 30% by weight to 40% by weight, the polyurethane being plasticizer-free. The fillers account for from 50% by weight to 70% by weight of the backing.

The isocyanate component of the polyurethane is selected in accordance with the specific properties to be established in the backing. Suitable examples include tolylene diisocyanate, diphenylmethane 4,4'-diisocyanate, dicyclohexylmethane 4,4'-diisocyanate, hexamethylene diisocyanate, isophorone diisocyanate, mixtures of the aforementioned isocyanates or isocyanates derived chemically therefrom, for example, dimerized or trimerized types.

The isocyanate-reactive component is likewise selected in accordance with the properties of the backing, which are to be established as a function of the desired profile of requirements. Suitable examples include all polyester diols, triols and polyols, polyether diols, triols and polyols, polyether diamines, triamines and polyamines, hydroxylfunctionalized polybutadiene, and also all monohydric alcohols (mono-ols), monofunctional amines (mono-amines), polyether mono-ols, polyether mono-amines, or products derived from the four last-mentioned groups.

It has been found advantageous if the hydroxyl-functionalized polybutadienes, the polyester diols, the polyester triols, the polyester polyols, the polyether diols, the polyether triols, the polyether polyols, the polyether diamines, the polyether triamines, or the polyether polyamines have a molecular weight  $M_w \ge 1000$  g/mol.

In addition to the isocyanate components recited above and the components which react with them, however, it is also possible to use other starting materials to form the polyurethane, without departing from the concept of the invention.

30

25

15

20

25

30

In order to accelerate the reaction between the isocyanate component and the isocyanate-reactive component, it is possible to use all catalysts known to the skilled worker, such as, for example, tertiary amines or organotin compounds.

5 Polyurethanes as described above are mentioned, for example, in "Ullmann's Encyclopedia of Industrial Chemistry, Vol. A21: Polyurethanes".

In one particularly preferred embodiment, an NCO/OH ratio of from 1.0 to 1.3 is established in order to form the polyurethane, preferably from 1.0 to 1.1.

The preferred mono-of OH content as a proportion of the overall OH content, i.e., the preferred chain-terminating component, is between 5% and 40%, in particular between 10% and 30%.

Fillers which may be used include both reinforcing types, such as carbon black, for example, and nonreinforcing types, such as chalk or barium sulfate, for example. Further examples are talc, mica, pyrogenic silica, silicates, zinc oxide, microballoons, solid glass microbeads, hollow glass microbeads, and/or plastic microbeads of all kinds. Mixtures of the materials mentioned may also be used.

The microballoons comprise elastic, thermoplastic hollow beads which have a polymer shell. These beads are filled with low-boiling liquids or with liquefied gas. Suitable shell polymers are, in particular, acrylonitrile, PVDC, PVC or acrylates. Hydrocarbons such as the lower alkanes, for example, pentane, are suitable as a low-boiling liquid, while a suitable liquefied gas is a chemical such as isobutane.

Particularly advantageous properties are in evidence when the microballoons involved are those having a diameter at 25°C of from 3  $\mu$ m to 40  $\mu$ m, in particular from 5  $\mu$ m to 20  $\mu$ m.

On exposure to heat, the capsules expand irreversibly and three-dimensionally. Expansion is at an end when the internal pressure equals the external pressure. In this way, a closed-cell foam backing is obtained which features good flow behavior and high recovery forces.

20

25

30

35

Following thermal expansion due to elevated temperature, the microballoons advantageously have a diameter of from 20  $\mu$ m to 200  $\mu$ m, in particular from 40  $\mu$ m to 100  $\mu$ m.

To enhance the stability of the polyurethane with respect to aging, it may be blended with customary aging inhibitors, which depending on the application in point may come from the class of the discoloring or nondiscoloring aging inhibitors, in the range between 0% by weight and 5% by weight, and also known light stabilizers, in the range between 0% by weight and 5% by weight, or ozone protectants, in the range between 0% by weight and 5% by weight.

To achieve freedom from bubbles, furthermore, it is possible to admix siccatives, such as calcium oxide or molecular sieve zeolites, for example, to the formulation, in particular in the range between 0% by weight and 10% by weight.

Depending on the intended use, all of the abovementioned auxiliaries may be used, either alone or in any desired combination, to prepare the polyurethane composition, in order to tailor it optimally to the use. The use of these additives also enables the composition to be colored black, as required in particular by the automotive industry, without problems.

The A and B components are either purchased directly or prepared from individual purchased components in accordance with customary mixing or preparation processes which accord with the prior art, for example, depending on rheological setting and filler content, in a stirred vessel, in a planetary mixer, or in a dissolver.

The particular feature of the process of the invention is the inverted nature of the coating operation.

Instead of the usual application of a pressure-sensitive adhesive composition to a backing, a reactive, initially liquid backing material is instead applied to a pressure-sensitive adhesive composition which has already been introduced, or, if appropriate, to other incoming materials, of which at least one has been treated with a pressure-sensitive adhesive composition.

One advantage of the process is to be able to achieve effective anchoring between the polyurethane backing material and the incoming, web-form materials, i.e., for example,

the pressure-sensitive adhesive composition, without the need to use primers or similar auxiliary layers. This is the case because, at the time of coating, the polyurethane backing material is reactive, owing to isocyanate which has not yet immediately reacted, and therefore undergoes spontaneous chemical attachment to numerous substrates.

5

10

Another advantage is the simplicity of the process, which makes it possible to produce self-adhesive articles in no more than three coating steps (preparation of the incoming material 1, preparation if appropriate of the incoming material 2, coating with the polyurethane backing material) with particular cost advantages, even when said articles are of complex construction, i.e., comprising a composite backing.

Following the coating operation with the two-component polyurethane, no further coating or laminating step is necessary to produce the self-adhesive article.

15

A further advantage of the process is the ability to produce a particular diversity of self-adhesive articles. This diversity arises from the diverse possibilities of polyurethane chemistry, as depicted above, and from the diverse possibilities in respect of the incoming materials 1 and 2.

20

The invention will be described more closely on the basis of the following examples, without wishing thereby to restrict the invention.

The following test methods were used to characterize briefly the specimens produced in accordance with the process described:

The bo

The bond strength was determined in accordance with BDF JOPMA002.
 In accordance with this method, the adhesive tape specimen for testing was applied to the substrate (steel) and then peeled off under defined conditions in a tensile testing machine. The peel angle was 180°, the peel speed 300 mm/min. The force required for peel removal is the bond strength.

30

25

The tensile strength and elongation at break were determined in the tensile test in accordance with BDF JOPMC001.

35 le

In this test, a test strip 100 mm in length and 25 mm in width was stretched in the lengthwise direction in a tensile testing machine at defined clamp speed (300 mm/min)

until it tore. The parameters measured were the tensile strength, based on the cross section of the sample, and the extension at the point of tear.

The compressive strength was determined in accordance with DIN 53577.

The compressive strength is the compression strain determined at a defined deformation (in the Examples 14%) during the stress operation. It was measured in a compressive testing machine. The dimensions of the test specimens were 30 mm x 30 mm x 15 mm (LxWxH). The height of the test specimens was produced by stacking the adhesive strips.

10

5

Coating in the examples was carried out on a unit from the company Pagendarm. The unit possessed the unwinding and winding facilities shown in Figure 1 for the incoming materials 1 and 2 with a web width of 50 cm. The coating gap width was variably adjustable between 0 and 1 cm. The length of the heating tunnel was approximately 12 m. The temperature in the heating tunnel was divisible into four zones and freely selectable in each case between room temperature and 120°C.

A two-component mixing and metering unit from the company Spritztechnik-EMC was used. The mixing system was dynamic. The mixing head was designed for two liquid and a third gaseous component. The mixing rotor had a variable speed of up to approximately 5000 rpm max. The metering pumps of this unit were toothed-wheel pumps having a maximum output of approximately 2 l/min.

25 The A components were prepared in an evacuable dissolver from the company Drais.

### Examples

30

# Example 1

To produce a special masking tape, which is used following the application of the first coat (cathodic electrocoat) to mask off the window flange joints during the coating

process in the OEM production of automobiles, and so protect them against further coats which are baked at temperatures of up to 180°C, the process was used as follows:

1st process step (preparation step), production of the incoming material 1:

5

A 23  $\mu$ m thick polyester film (polyethylene terephthalate) was coated in a customary coating process with a known natural rubber-based pressure-sensitive adhesive composition comprising

	48%	natural rubber CV50
10	23%	poly-beta-pinene resin
	5%	terpene phenolic resin
	3%	rosin
	7%	copolymer of acrylonitrile and butadiene
	8%	zinc oxide
15	5%	reactive alkylphenol resin, and
	1%	2,5 di(tert-amyl)hydroquinone

from a solution in an application thickness of approximately 25  $\mu$ m and, during winding, was lined with a standard commercial release paper.

20

25

2nd process step, polyurethane coating:

In the 2nd process step, the polyester film treated with the pressure-sensitive adhesive composition was coated from the nonadhesive side with a devolatilized, two-component polyurethane backing composition at a rate of 1 m/min. The application thickness was  $120 \, \mu \text{m}$ . Curing was effected at a tunnel temperature of  $80^{\circ}\text{C}$ .

The makeup of the polyurethane backing composition was as follows:

15

	Raw material	Weight fraction [% by weight]
	Arcol 1030 ®	30.0
A component	Arcol 1074 ®	10.0
	Dibutyltin dilaurate	0.2
	Calcium oxide	5.0
	Bayferrox 3920 ®	1.0
	Omyacarb 4BG ®	28.3
B component	Vestanat IPDI ®	25.5

The resultant adhesive tape had a tensile strength of 30.3 N/mm<sup>2</sup> with an elongation at break of 43.4%. The bond strength on steel was 4.9 N/cm.

The adhesive tape was overpaintable and was sufficiently temperature-stable in view of the paint baking conditions.

# Example 2

To produce a special masking tape, which is used as in Example 1 following the application of the first coat (CED coating material) to mask off the window flange joints during the coating process in the OEM production of automobiles, and so protect them against further coats which are baked at temperatures of up to 180°C, and which is also so flexible that it can easily be stuck on in curves, the process was used as follows:

1st process step (preparation step), production of the incoming material 1:

A solvent-based acrylate pressure-sensitive adhesive composition consisting of butyl acrylate (47.5%), ethylhexyl acrylate (47.5%), glycidyl methacrylate (2%), acrylic acid (3%), and small amounts of a known crosslinker was applied in an application thickness of 40 g/m² to standard commercial double-sided release paper, dried, crosslinked and subsequently wound up.

2nd process step, polyurethane coating:

In the 2nd process step, the acrylate pressure-sensitive adhesive composition applied to the release paper was coated directly with a devolatilized, two-component polyurethane backing composition at a rate of 3 m/min. The application thickness was 250  $\mu$ m. Curing was effected at a tunnel temperature of 60 to 70°C.

The makeup of the polyurethane backing composition was as follows:

	Raw material	Weight fraction
		[% by weight]
	Poly THF 250 ®	14.3
A component	Poly THF 650 ®	37.6
	Dibutyltin dilaurate	0.1
	Calcium oxide	10.0
	Bayferrox 3920 ®	1.3
	Aerosil R 202 ®	2.0
B component	Desmodur CD ®	34.7

The resultant adhesive tape had a tensile strength of 20.0 N/mm<sup>2</sup> with an elongation at break of 195%. The bond strength on steel was 2.5 N/cm. The adhesive tape was overpaintable and was sufficiently temperature-stable in view of the paint baking conditions, and could be stuck on in curves.

15 Example 3

To produce an elastic, sandblast-resistant, punchable adhesive stencil tape, the process was used as follows:

20 1st process step (preparation step), production of the incoming material 1:

A solvent-based acrylate pressure-sensitive adhesive composition consisting of butyl acrylate (47.5%), ethylhexyl acrylate (47.5%), glycidyl methacrylate (2%), acrylic acid (3%), and small amounts of a known crosslinker was applied in an application thickness

5

of 40 g/m<sup>2</sup> to standard commercial double-sided release paper, dried, crosslinked and subsequently wound up.

2nd process step, polyurethane coating:

In the 2nd process step, the acrylate pressure-sensitive adhesive composition applied to the release paper was coated directly with a devolatilized, two-component polyurethane backing composition at a rate of 3 m/min. The application thickness was 875  $\mu$ m. Curing was effected at a tunnel temperature of 60 to 70°C.

10 The makeup of the polyurethane backing composition was as follows:

	Raw material	Weight fraction
		[% by weight]
	Arcol 1042 ®	29.0
A component	Dibutyltin dilaurate	0.1
	Omyacarb 4BG ®	63.1
	Calcium oxide	5.0
B component	Desmodur CD ®	2.8

The resultant adhesive tape had a tensile strength of 1.7 N/mm<sup>2</sup> with an elongation at break of 124%. The bond strength on steel was 2.5 N/cm. The adhesive tape was sufficiently resistant to sandblasting, and was readily punchable.

# Example 4

To produce a sandblast-resistant, punchable adhesive stencil tape of low extensibility at low applied force, the process was used as follows:

1st process step (preparation step), production of the incoming material 1:

A solvent-based acrylate pressure-sensitive adhesive composition consisting of butyl acrylate (47.5%), ethylhexyl acrylate (47.5%), glycidyl methacrylate (2%), acrylic acid (3%), and small amounts of a known crosslinker was applied in an application thickness

15

of 40 g/m<sup>2</sup> to standard commercial slightly creped paper backing with a basis weight of 68 g/m<sup>2</sup>, dried, crosslinked and, during winding up, was lined with a standard commercial release paper.

2nd process step, polyurethane coating:

In the 2nd process step, the slightly creped paper backing treated with the pressure-sensitive adhesive composition was coated from the nonadhesive side with a devolatilized, two-component polyurethane backing composition at a rate of 3 m/min. The application thickness was 850  $\mu$ m. Curing was effected at a tunnel temperature of 60 to 70°C.

The makeup of the polyurethane backing composition was as follows:

	Raw material	Weight fraction [% by weight]
	Arcol 1042 ®	29.0
A component	Dibutyltin dilaurate	0.1
	Omyacarb 4BG ®	63.1
	Calcium oxide	5.0
B component	Desmodur CD.®	2.8

The resultant adhesive tape was sufficiently resistant to sandblasting, and was readily punchable. The bond strength on steel was 2.1 N/cm.

20 Example 5

To produce a sandblast-resistant, punchable adhesive stencil tape of low extensibility at low applied force, the process was used as follows:

25 1st process step (preparation step), production of the incoming material 1:

15

A solvent-based acrylate pressure-sensitive adhesive composition consisting of butyl acrylate (47.5%), ethylhexyl acrylate (47.5%), glycidyl methacrylate (2%), acrylic acid (3%), and small amounts of a known crosslinker was applied in an application thickness of 40 g/m $^2$  to a standard commercial polyester (polyethylene terephthalate) film with a thickness of 23  $\mu$ m, dried, crosslinked and, during winding up, was lined with a standard commercial release paper.

2nd process step, polyurethane coating:

In the 2nd process step, the polyester film treated with the pressure-sensitive adhesive composition was coated from the nonadhesive side with a devolatilized, two-component polyurethane backing composition at a rate of 3 m/min. The application thickness was  $850 \, \mu \text{m}$ . Curing was effected at a tunnel temperature of 60 to  $70^{\circ}\text{C}$ .

The makeup of the polyurethane backing composition was as follows:

	Raw material	Weight fraction
		[% by weight]
	Arcol 1042 ®	29.0
A component	Dibutyltin dilaurate	0.1
	Omyacarb 4BG ®	63.1
	Calcium oxide	5.0
B component	Desmodur CD ®	2.8

The resultant adhesive tape was sufficiently resistant to sandblasting, and was readily punchable. The bond strength on steel was 2.8 N/cm.

# Example 6

To produce an elastic masking tape which is easy to stick on in curves and is suitable for general painting and decorating work, the process was used as follows:

1st process step (preparation step), production of the incoming material 1:

20

25

15

A solvent-based acrylate pressure-sensitive adhesive composition consisting of butyl acrylate (47.5%), ethylhexyl acrylate (47.5%), glycidyl methacrylate (2%), acrylic acid (3%), and small amounts of a known crosslinker was applied in an application thickness of 40 g/m² to standard commercial double-sided release paper, dried, crosslinked and then wound up.

2nd process step, polyurethane coating:

In the 2nd process step, the acrylate pressure-sensitive adhesive composition applied to the release paper was coated directly with a devolatilized, two-component polyurethane backing composition at a rate of 3 m/min. The application thickness was 300  $\mu$ m. Curing was effected at a tunnel temperature of 60 to 70°C.

The makeup of the polyurethane backing composition was as follows:

	Raw material	Weight fraction [% by weight]
· · · · · · · · · · · · · · · · · · ·	Arcol 1074 ®	31.5
A component	Lutensol A07 ®	3.3
	Dibutyltin dilaurate	0.1
	Calcium oxide	7.9
	Omyacarb 4BG ®	53.9
B component	Desmodur CD ®	3.3

The resultant adhesive tape had a tensile strength of 2.1 N/mm<sup>2</sup> with an elongation at break of 194%. The bond strength on steel was 2.5 N/cm. The adhesive tape was overpaintable and could be stuck on in curves.

### Example 7

To produce an adhesive edge-protection tape, the process was used as follows:

25 1st process step (preparation step), production of the incoming material 1:

20

15

A solvent-based acrylate pressure-sensitive adhesive composition consisting of butyl acrylate (47.5%), ethylhexyl acrylate (47.5%), glycidyl methacrylate (2%), acrylic acid (3%), and small amounts of a known crosslinker was applied in an application thickness of  $40 \text{ g/m}^2$  to a woven backing made from closely woven polyester ( $20 \times 20 \text{ threads per cm}$  in warp and weft direction, metric count: 34, basis weight > 130 g/m²), dried, crosslinked and, during winding up, was lined with a standard commercial release paper.

2nd process step, polyurethane coating:

In the 2nd process step, the woven fabric treated with the pressure-sensitive adhesive composition was coated from the nonadhesive side with a devolatilized, two-component polyurethane backing composition at a rate of 3 m/min. The application thickness was  $400 \ \mu m$ . Curing was effected at a tunnel temperature of  $60 \ to \ 70^{\circ}C$ .

The makeup of the polyurethane backing composition was as follows:

	Raw material	Weight fraction [% by weight]
	Arcol 1042 ®	29.0
A component	Dibutyltin dilaurate	0.1
	Omyacarb 4BG ®	63.1
	Calcium oxide	5.0
B component	Desmodur CD ®	2.8

The resultant adhesive tape had a tensile strength of 19.1 N/mm<sup>2</sup> with an elongation at break of 24%. The bond strength on steel was 2.9 N/cm.

20

#### Example 8

To produce a particularly cost-effective adhesive edge-protection tape, the process was used as follows:

25 1st process step (preparation step), production of the incoming material 1:

15

20

25

A solvent-based acrylate pressure-sensitive adhesive composition consisting of butyl acrylate (47.5%), ethylhexyl acrylate (47.5%), glycidyl methacrylate (2%), acrylic acid (3%), and small amounts of a known crosslinker was applied in an application thickness of  $40 \text{ g/m}^2$  to a  $150 \,\mu\text{m}$  thick spunbonded polyester nonwoven with a basis weight of  $70 \text{ g/m}^2$ , dried, crosslinked and, during winding up, was lined with a standard commercial release paper.

2nd process step, polyurethane coating:

In the 2nd process step, the spunbonded nonwoven treated with the pressure-sensitive adhesive composition was coated from the nonadhesive side with a devolatilized, two-component polyurethane backing composition at a rate of 3 m/min. The application thickness was 400  $\mu$ m. Curing was effected at a tunnel temperature of 60 to 70°C.

The makeup of the polyurethane backing composition was as follows:

	· Raw material	Weight fraction
	·	[% by weight]
	Arcol 1042 ®	29.0
A component	Dibutyltin dilaurate	0.1
	Omyacarb 4BG ®	63.1
	Calcium oxide	5.0
B component	Desmodur CD ®	2.8

The resultant adhesive tape had a tensile strength of 9.8 N/mm<sup>2</sup> with an elongation at break of 18%. The bond strength on steel was 2.6 N/cm.

# Example 9.

To produce an adhesive printing plate mounting strip for the printing industry, the process was used as follows:

1st process step (preparation step), production of the incoming material 1:

25

A solvent-based acrylate pressure-sensitive adhesive composition consisting of ethylhexyl acrylate (70%), stearyl acrylate (17%), acrylic acid (3%), and known resins (10%) was applied in an application thickness of 60 g/m² to standard commercial double-sided release paper, dried and subsequently wound up.

5

2nd process step (preparation step), production of the incoming material 2:

Process step 1 was repeated.

10 3rd process step, polyurethane coating:

Between the acrylate pressure-sensitive adhesive compositions from process steps 1 and 2, applied to the release papers, coating took place with a devolatilized, two-component polyurethane backing composition at a rate of 1 m/min. The application thickness was 1.5 mm. Curing was effected at a tunnel temperature of 90°C.

The makeup of the polyurethane backing composition was as follows:

	Raw material	Weight fraction [% by weight]
	Arcol 1042 ®	41.0
	Arcol 1043 ®	41.0
A component	Dibutyltin dilaurate	0.2
	Kronos 2160 ®	1.6
	Calcium oxide	5.0
	Aerosil R 202 ®	3.5
B component	Vestanat IPDI ®	7.7

The resultant adhesive tape had a compressive strength  $H_{14}$  of 22 N/cm<sup>2</sup>. The bond strength on steel was 3.4 N/cm.

# Example 10

To produce a microballoon-foamed adhesive printing plate mounting strip for the printing industry, the process was used as follows:

10

15

20

1st process step (preparation step), production of the incoming material 1:

A solvent-based acrylate pressure-sensitive adhesive composition consisting of ethylhexyl acrylate (70%), stearyl acrylate (17%), acrylic acid (3%), and known resins (10%) was applied in an application thickness of 60 g/m<sup>2</sup> to standard commercial double-sided release paper, dried and subsequently wound up.

2nd process step (preparation step), production of the incoming material 2:

Process step 1 was repeated.

3rd process step, polyurethane coating:

Between the acrylate pressure-sensitive adhesive compositions from process steps 1 and 2, applied to the release papers, coating took place with a devolatilized, two-component polyurethane backing composition at a rate of 1 m/min. The application thickness was 1.5 mm. Curing was effected at a tunnel temperature of 90°C.

The polyurethane backing composition contained preexpanded thermoplastic hollow beads (Expancel ®) and its makeup was as follows:

	Raw material	Weight fraction
		[% by weight]
	Arcol 1042 ®	39.5
	Arcol 1043 ®	39.5
A component	Dibutyltin dilaurate	0.2
	Kronos 2160 ®	1.9
	Calcium oxide	5.0
	Expancel 551 DE 80 ®	3.0
	Aerosil R 202 ®	3.5
B component	Vestanat IPDI ®	7.4

The resultant adhesive tape had a compressive strength  $H_{14}$  of 30 N/cm<sup>2</sup>. The bond strength on steel was 2.9 N/cm.

5

#### Example 11

To produce a nitrogen-foamed adhesive printing plate mounting strip for the printing industry, the process was used as follows:

10 1st process step (preparation step), production of the incoming material 1:

A solvent-based acrylate pressure-sensitive adhesive composition consisting of ethylhexyl acrylate (70%), stearyl acrylate (17%), acrylic acid (3%), and known resins (10%) was applied in an application thickness of 60 g/m<sup>2</sup> to standard commercial double-sided release paper, dried and subsequently wound up.

2nd process step (preparation step), production of the incoming material 2:

Process step 1 was repeated.

20

25

15

3rd process step, polyurethane coating:

Between the acrylate pressure-sensitive adhesive compositions from process steps 1 and 2, applied to the release papers, coating took place with a devolatilized, two-component polyurethane backing composition at a rate of 1 m/min. Nitrogen was introduced into the polyurethane backing composition directly at the mixing head, so that the cured backing had a density of 0.7 g/cm<sup>3</sup>. The application thickness was 1.5 mm. Curing was effected at a tunnel temperature of 90°C.

The makeup of the polyurethane backing composition was as follows:

	Raw material	Weight fraction [% by weight]
	Arcol 1030 ®	17.0
	Arcol 1067S ®	40.0
A component	Dibutyltin dilaurate	. 0.2
	Kronos 2160 ®	2.4
	Calcium oxide	9.0
	Aerosil R 202 ®	3.5
B component	Vestanat IPDI ®	27.9

The resultant adhesive tape had a compressive strength  $H_{14}$  of 28 N/cm<sup>2</sup>. The bond strength on steel was 2.7 N/cm.

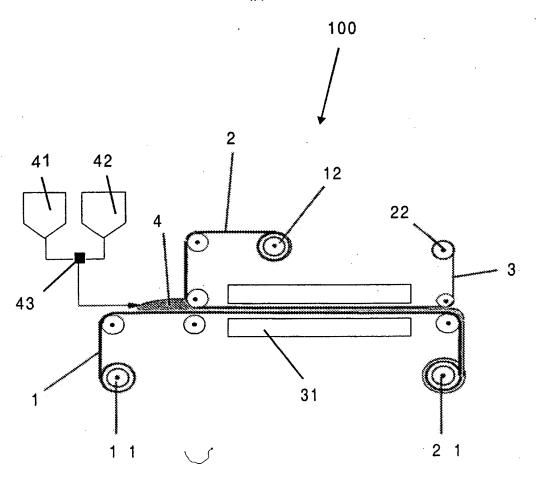


Fig . 1